MINISTÈRE DE L'ENVIRONNEMENT
ET DE LA LUTTE CONTRE
LES CHANGEMENTS CLIMATIQUES

Fertilizing Residual Materials Sampling Protocol and Special Accreditation Provisions (DR-12-MRF-02)

Updated to February 22, 2022

Votre gouvernement

Québec

Coordination and editing
Prepared by the Centre d'expertise en analyse environnementale du Québec (Ministère de l'Environnement et de la Lutte contre les changements climatiques–MELCC) and produced by the MELCC Direction des communications

Information

Telephone numbers: 418-521-3830
1-800-561-1616 (toll free)

Fax: 418-646-5974
Form: www.environnement.gouv.qc.ca/formulaires/renseignements.asp
Internet: www.environnement.gouv.qc.ca

Copies are available from the
Centre d'expertise en analyse environnementale du Québec (Ministère de l'Environnement et de la Lutte contre les changements climatiques)
2700, rue Einstein, bureau E-2-220
Québec (Québec) G1P 3W
Telephone: 418-643-1301
Fax: 418-528-1091
Email: ceaeq@environnement.gouv.qc.ca

Or

On the CEAEQ section of the www.environnement.gouv.qc.ca website or at www.ceaeq.gouv.qc.ca.

Legal Deposit–2022
Bibliothèque et Archives nationales du Québec
ISBN 978-2-550-91750-2 (PDF), English Edition: 2022
ISBN 978-2-550-90542-4 (PDF), French Edition: 2021

Table of Contents

Introduction

This publication describes the sampling procedure for industrial and municipal solid/sludge fertilizing residual materials (hereinafter “FRM”). Its goal is to standardize sampling practices for FRM that could subsequently be subject to reclamation under regulations and per manuals stemming from the *Environment Quality Act* (EQA) that are in effect at the Ministère de l’Environnement et de la Lutte contre les changements climatiques. Special provisions applicable to fertilizing residual materials sampling for chemical, microbiological and foreign matter analysis are also defined herein.

The use of this protocol is also part and parcel of accreditation requirements for applicants to the Programme d’accréditation d’échantillonnage environnemental (PAEE), in compliance with the stipulations set out in the *Processus et exigences d’accréditation - Matières résiduelles fertilisantes - Secteur agricole* (DR-12-MRF).

Finally, the publication is addressed to anyone with an interest in improving the quality of samples collected for the characterization of fertilizing residual materials. Accredited firms whose sampling practices comply with the framework of the accreditation program are also required to employ a protocol matching the requirements of the Centre d’expertise en analyse environnementale du Québec (CEAEQ), and follow the guidelines and principles set out herein, even if their procedures are certified in compliance with a Bureau de normalisation du Québec (BNQ) standard.

Limitation

This publication does not cover liquid residue sampling.[i]

Definitions

Note: Most types of recovered solid FRM in Québec are listed in the glossary section of the *Guide sur le recyclage des matières résiduelles fertilisantes* (Guide to fertilizing residual materials recycling–available in French only).

Firm–a company, cooperative or legal person, as defined in the EQA.

Lot–the total quantity of a more or less homogeneous material deemed to have uniform characteristics and manufactured under uniform conditions.

Sampling location–general location where sampling will be carried out: can be an address, a description or be identified by means of maps, aerial photos, specifications, etc.

Sampling point–name given to the exact location where a sample—or portion thereof—is collected. For example, the outlet of a sludge press may have one or more sampling points. Another example would be the end point of the conveyor belt where a sample is collected. It is also the name of the column or a hole dug into a pile when sampling a batch-produced FRM is called for.

Sampling site–a more detailed description or definition of the location where sampling is carried out. For example, this could be the location of the treatment facility room containing the sludge press, conveyor belt or other equipment and where sampling is conducted (may also refer to the pile or other location selected for sampling).

FRM production

Batch production–a process by which predetermined quantities of FRM are produced and accumulated or stored in piles or batches. Batch production can be segmented in time or space and also involve the production of relatively small individual quantities.

Continuous production–a process by which FRM are produced by the flow of matter over a specific period of time, without stoppage.

1. General information

1.1 Specific provisions related to accreditation

1.1.1 Sampling data log

Keeping an orderly sampling data log of activities and that relates all relevant operational sampling facts is essential (see the template in Appendix II). The log must describe the sampling method used or refer to it and stipulate the collection equipment that was used. The individual that conducted sampling must in each case record the following, and sign the log:

- The sample identification number;
- The location, date and time of collection;
- The sampling point;
- The collection equipment and supplies used;
- The type of residue and analyses requested;
- The temperature in the cooler;
- The performed analyses;
- The sampler's identification information.

Moreover, all special information, such as weather and/or change in sampling method, must be recorded. Appendix II offers a sampling data log template.

1.1.2 Error correction

1.1.2.1. Correcting previously recorded data

Data errors must be individually crossed out by a single line and not erased, made illegible or otherwise removed, and the correct value written in next to them. All such data changes must be signed or initialled and dated by the individual making the correction. Equivalent measures are required for electronically stored records to avoid any original data losses or modifications.

1.1.2.2. Error correction in reports

Post-submission corrections, changes or additions to a technical report must always include either a new report or data transfer and identified as "Supplemental sampling report number… or other stipulation… [or other similar formulation]. In cases where a new technical report is called for, it must both have a unique identifier and mention the original report it replaces. The client must be informed about any changes or amended sections.

1.1.3 Internal audits

The science officer is responsible for planning and conducting the annual internal technical and management audits. Technical audits must be conducted at the sampling points used by the individual samplers and cover all types of sampling and analysis described herein.

The annual management system audits must cover all aspects, including documentation review, staff training, record keeping, quality control, etc. The goal is to check whether the firm's activities continue to comply with the requirements set out herein or in the *Processus et exigences d'accréditation – Matières résiduelles fertilisantes – Secteur agricole* (DR-12-MRF).

The science officer must prepare the annual internal audit plan covering all aspects of sampling and/or accreditation scope and samplers over a two-year period.

Audit reports must be kept and the science officer is responsible for follow-up in order to take compliance corrective measures found during the audit process and review opportunities for improvement.

1.2 Special technical aspects

1.2.1 Protective equipment

Samplers must always wear disposable latex or nitrile gloves and change them when necessary whenever conducting sampling.

Samplers must take all appropriate health and safety measures when handling FRM likely to contain pathogens or to release bioaerosols or dust. Appendix I lists the pathogen preventive measures that are mandatory for workers that handle FRM.

1.2.2 Sample types

FRM analysis is made from spot samples or composites for microbiology parameters and composite samples for chemical and foreign matter parameter analysis.

Representative spot samples in dynamic settings are taken over a short interval of time, usually less than 15 minutes.

Representative grab samples in static settings are taken at a specified location or in a particular lot.

Composite samples are comprised of either spot or grab samples in equal proportions or in proportion to their representative weights, volumes or lots. Composite samples can be prepared at the sampling site itself, using an appropriate container that is sufficient for ensuring full homogenization of the final sample, without any loss.

The first step is to collect each of the grab or spot samples using the same sampling method, mix them thoroughly in the container to form a single sample, and transfer it to a suitable container for storage and transport to the laboratory. When the volume of the aggregate grab or spot samples is greater than 10 litres, the composite sample should be prepared by dividing it using the quartering technique.

2. Sampling for foreign matter analysis

When foreign matter analysis is required, the equipment selected by the firm for sampling can be adapted on the basis of FRM type, using adequate safety precautions. Sampling for foreign matter analysis can be combined with any other type of analysis, since no danger of contamination exists for this parameter. The equipment used must enable representative sampling of the matter as a whole during the procedure, whether production is continuous or in batches. In some cases, sampling for foreign matter may use inorganic chemistry parameters for both continuous and batch processes. Regardless of which other type of analysis foreign matter sampling is paired with, the volume of spot or grab samples must be two litres.

Exceptionally, when analyzing samples of fallen leaves and source-sorted green plant residues (plant matter) for foreign matter, a sample of five litres is required.

The final volume of the composite sample is reduced using the quarterage technique, obtaining a volume of less than 10 litres in order to fill a two-litre sample container (five litres for plant matter). Either a sufficient number of smaller coolers are required, or a single cooler whose volume is sufficient to hold all samples collected in the sampling campaign.

3. Sampling for chemical parameter analysis

General information

Analytical requirements are set out in Table 6.1 of the *Guide sur le recyclage des matières résiduelles fertilisantes*. However, when sampling matter certified compliant with a BNQ standard, additional tests not mentioned in the guide will be needed, per the requirements of the applicable standard.

For the analysis of chemical parameters, a composite sample should be prepared using spot samples for continuous production and grab samples for batch production. In the case of continuously produced FRM, the sampling campaign takes place over a representative period of time of the facility's and FRM production. Spot samples of identical volume are withdrawn at regular intervals. At least 8 spot samples must be taken at 60-minute intervals over 24 hours of production. For production over less than 24 hours and if sampling at 60-minute intervals is not feasible, the 8 samples should be taken at regular intervals over the entire time span of production.

The following directive applies to sample conservation between the time of withdrawal and receipt by the laboratory: "Do not freeze the samples, and adjust the number, volume and position of freezer packs on the basis of the number, mass and initial temperature of the samples, to cool them." Samples must be delivered to the laboratory with all due haste.

Disposable or veterinary gloves can be used to homogenize materials with lumps, clumps or portions that are difficult to break up with a ladle, spoon or other tool. However, the following precautions are mandatory:

- Disposable gloves are to be preferred. If reusable gloves are used more than once, they must be cleaned (3.1.2), and decontaminated (3.2.2) for organic analysis or disinfected for microbiology analysis (4.1.2);
- No part of a sample can touch a surface that has not already been cleaned, decontaminated or disinfected;
- The entire length of the glove must cover the arm of the sampler up to the shoulder to avoid contamination from clothing or skin.

Veterinary gloves are quite fragile and easily torn. Additional measures are therefore required to prevent sample rejection caused by a torn glove. For example, a latex glove worn over or under a veterinary glove would be an adequate preventive measure.

Veterinary gloves are never authorized for special cases of analysis of other organic parameters such as polycyclic aromatic hydrocarbons (PAHs), volatile organic compounds (VOCs) and semi-volatile organic compounds (SVOCs), phenolic compounds and formaldehyde levels. In these cases, clean decontaminated metal tools or instruments are used for sampling all types of FRM and for the homogenization of composite samples.

3.1 Sampling continuously produced fertilizing residual matter for inorganic parameter analysis [1]

Samples must be taken using the method set out in section 3.1.4.

3.1.1 Required supplies

- A clean plastic bucket with a lid, approximate usable volume of about 20 litres;
- One- or two-litre, wide mouth graduated plastic or glass sampling containers with lids, when required[2];
- One- or two-litre, wide mouth plastic or glass sample jars for shipping to the laboratory;
- One- or two-litre sealable or slide closure bags if sample jars are not used;
- A clean spoon or ladle;
- Frozen freezer packs;
- A cooler whose volume is sufficient to contain the sample and the appropriate number of freezer packs;
- Plastic sheeting, as needed;
- A plastic scoop, as needed;
- A thermometer (for batch use if there is more than one composite sample);
- Latex or nitrile disposable gloves (disposable or reusable veterinary gloves if used);
- Soapy water, rinse water (optional), distilled water and clean rags.

3.1.2 Cleaning supplies

All reusable supplies must be precleaned with soapy water then rinsed with tap water (optional), rinsed again with distilled water and dried in the open air (unless environmental recontamination is suspected) or with a clean rag. All supplies used for sampling must be stored in a clean area or in a container that is protected from contamination.

3.1.3 Labelling supplies

All buckets, jars and bags must be labelled prior to each sampling period, with numbers that identify the sampling site and test type. The bucket, jar and/or bag used at a single sampling site must bear the same

[1] These parameters may include total neutralizing value and total phosphorus (P_2O_5) when necessary.

[2] Minimum sampling volume is permitted when there are no other requirements. Foreign matter sampling requires 2-litre samples. The capacity of all other containers must be adjusted in the case of foreign matter sampling if the sampler wishes to combine withdrawals for multiple parameters.

number as the container sent for analysis. When sampling continuously produced FRM, the cooler may be labelled rather than the bucket, if there is only one bucket and a single composite sample for the entire sampling campaign.

In cases where there is only one sampling point and no real possibility of confusing the sampling containers, labelling is not mandatory.

3.1.4 Sampling method

Where FRM are discharged into multiple outlet channels, they all must be sampled in equivalent manner during the same day.

A one-litre spot sample is taken at the outlet of the sampling point with an appropriate instrument. If deemed safe, solid matter may be manually sampled using fresh disposable gloves. Sampling containers without lids are acceptable to the extent that the samples are transferred immediately and there is no danger of airborne contamination. Each one-litre spot sample is transferred to an approximately 20-litre plastic bucket as soon as it is taken for the duration of the sampling campaign, the bucket being kept at approximately 6°C. Temperature in the cooler must be checked and noted hourly or every time a sample is taken. The thermometer must never be placed in contact with the freezer packs but kept in the bucket containing the sample or in a representative section of the temperature surrounding the sample inside the cooler.

Once all samples have been collected and loaded into the bucket, the composite sample is homogenized using a plastic spoon or ladle. Homogenization may also be performed using disposable or veterinary gloves (see section 3).

If the volume of the sample is equal to or less than 10 litres, a fraction of one litre of the composite sample is transferred into a sealable container that is already labelled and ready to ship to the lab using the method described in section 3.4. The volume of the sample must be adjusted when using plant matter for foreign matter analysis.

If the volume of the sample in the bucket exceeds 10 litres, the quarterage technique is used to divide it. This means emptying out the contents of the bucket onto a plastic sheet or other suitable surface such as glass, Teflon, Plexiglas, etc. The quarterage surface must be composed of a material that can withstand mixing by a scoop or sampling tool. The surface area must be sufficient to accommodate the materials and allow for mixing without any overflow. A pile of regular shape is then made using a plastic scoop, and divided into four. Two opposing quarters are then discarded and the remaining two combined. The process is repeated until a composite sample of the desired size (less than 10 litres) is obtained. The composite sample is then transferred to a previously labelled sealable plastic or glass container that is ready to be shipped to the laboratory using the method described in section 3.4.

3.2 Sampling continuously produced FRM for organic parameter analysis

Per the *Guide sur le recyclage des matières résiduelles fertilisantes,* dry analysis of the percentage of organic matter is frequently required. Dioxins and furans are also organic chemistry parameters that are frequently needed for the characterization of certain FRM residues.

Other parameters may be required in special cases of high risk of contamination by PAHs, volatile organic compounds (VOCs) and semi-volatile organic compounds (SVOCs), phenolic compounds, formaldehyde and petroleum hydrocarbons (C_{10}-C_{50}). In such cases, using any type of glove is prohibited when handling, sampling and homogenizing a sample. A metal spoon or ladle is required to avoid contamination by plastic, and equipment decontamination is crucial in such cases.

Note: PAHs, dioxins and furans are light-sensitive. As such, samplers need to take precautions to protect samples from light.

Samples must be taken using the method set out in section 3.2.4.

3.2.1 Required supplies

- A clean metal bucket with a metal lid, approximate 20-litre capacity;
- A wide-mouth graduated amber glass, metal or aluminum-covered lidded glass sample container, as needed;
- Aluminum foil or Teflon film for the lid, as needed;
- A clean metal spoon or ladle;
- Frozen freezer packs;
- A cooler of sufficient size to contain the sample and several freezer packs;
- A one litre, wide mouth, amber glass or aluminum-covered lidded glass for shipment to the laboratory;
- A metal scoop, as needed;
- A metal, Teflon or glass surface, as needed;
- A thermometer;
- Latex or nitrile disposable gloves;
- Soapy water, rinse water (optional), distilled water and clean rags;
- Acetone, hexane, and a recovery canister.

3.2.2 Cleaning and decontamination

All sampling supplies must be clean and available on the sampling site. Prior off-site cleaning and decontamination is suggested but can also be handled on-site. As stated in the *Guide d'échantillonnage à des fins d'analyses environnementales, Cahier 1 – Généralités*, the number and type of cleaning agents does not guarantee effective cleaning. Instead, carefully following procedure by the sampler at each stage of decontamination is vital.

Mandatory procedure:

- Wash with soapy water;
- Tap water rinse (optional);
- Distilled water rinse;
- Acetone rinse;
- Two separate hexane rinses;
- Acetone rinse;
- Open-air drying.

Acetone and hexane rinsing must be performed in a way that sufficiently moistens the surface with solvent so as to dissolve and eliminate, through contact, all traces of organic contaminants that may have adhered to the surface of the metal. In order to properly rinse the entire surface of the material used, the solvent must be seen to trickle.

However, if this type of decontamination cannot be performed safely due to the setup of the plant, it may be possible to conclude an agreement with the lab that is to perform the analyses for it to provide the required sampling supplies and handle decontamination.

All supplies used for sampling must be stored at an appropriate location or in a contamination-free metal container.

3.2.3 Labelling supplies

Buckets and jars must be labelled prior to sampling, with numbering that is associated with the sampling point and type of analysis required. Labelling must use the same number as the container sent for analysis. In the case of continuous sampling, the cooler (rather than the bucket) may be tagged if it contains only one bucket and a single composite sample for the entire sampling campaign.

If a single sampling point is used and there is no real danger of confusion, the sampling containers need not be labelled.

3.2.4 Sampling method

A one-litre spot sample is taken at the outlet of the sampling point, using a metal spoon or ladle. If deemed safe, solid matter may also be removed manually using new disposable gloves. The sample is then placed in an appropriate lidded container so as to standardize all samples to a volume of one litre. Non-lidded containers are acceptable if sample transfer is immediate and there is no danger of airborne contamination. All one-litre spot samples are transferred to a metal bucket of approximately 20 litres and stored at approximately 6°C throughout the entire sampling campaign. The temperature in the cooler must be checked and noted hourly or whenever a sample is taken. Thermometers must never be in direct contact with the freezer packs but rather remain in the sample bucket or section that is representative of the temperature inside the cooler.

Once all samples have been taken and placed in an appropriate container, the composite sample is homogenized using the metal ladle. When an analysis of the percentage of organic matter and dioxins and furans is called for, disposable or veterinary gloves can be worn to effect homogenization (see section 3).

If sample volume is less than or equal to 10 litres, a one litre fraction of the composite sample is transferred to an appropriate, prelabelled glass container. The mouth of the jar is covered with aluminum foil or Teflon film (if the lid does not already include this feature) and the lid is tightly twisted down. The container is now ready for shipping to the laboratory using the method described in section 3.4.

If the volume of the sample exceeds 10 litres, the quarterage technique is used to divide it. This means emptying out the contents of the bucket onto a suitable surface that is sufficient in size to allow for mixing without overflow. A pile of regular shape is made using a metal scoop, and divided into four. Two opposing quarters are then discarded, and the remaining two combined. The process is repeated until a composite sample of the desired size is obtained. One litre of the composite sample is then transferred to a previously labelled, sealable container. If organic composite analysis is planned, aluminum foil or Teflon film must also be used to seal the lid of the container.

3.3 Batch FRM sampling

Batch FRM sampling is required when continuous production is irregular, uses uncommon procedures or when the planned sampling point is inaccessible or not secure.

Priority should be given to continuously produced FRM production.

The following protocol applies to batch FRM. Depending on the parameters to be analyzed, the sampler will select the supplies, labelling and cleaning and decontamination methods described in section 3.1 or 3.2. However, grab samples must be collected using the method described in section 3.3.1.

3.3.1 FRM pile sampling method

At least 10 grab samples are needed for the composite sample for piles whose volume is less than 400 m³. If volume exceeds that figure, the appropriate number of samples is determined by the following formula:

$$n = \frac{\sqrt{V}}{2}$$

where n is the number of samples and V is the volume in cubic metres (m³). The number of samples should not exceed 30.

The 30-sample maximum applies to piles with a maximum volume of 3,600 m³.If the pile has a volume greater than 3600 m3, it must be split evenly into sections, theoretically or virtually. The determination of the right number of grab samples uses the same formula for each section. For example, for a 5,000 m³ pile, the pile is divided into two separate 2 500 m³ (< 3 600 m³) sections. Twenty-five (25) grab samples must be taken to prepare a composite sample for each of the sections. In this example, two composite samples will be tested.

Sampling must be representative of the pile. As such, the complete pile must be covered, for example by quartering into a number of sections that corresponds to the previously determined number of grab samples. Sampling must take place on a volume of matter sufficient to enable evaluation of the characteristics of the matter that will be recovered.

Once all the sampling columns or holes are established and are representative of the pile matter, it can be used to withdraw samples for all required analyses, while carefully following the correct individual protocols described herein.

Grab sample volumes must be identical and minimally between 0.5 and 1 litre[3], taken at a depth of between 30 centimetres and 1 metre and alternating from the top, middle and bottom of the pile. Grab samples must be taken using an appropriate tool (see the specifications for organic and/or inorganic parameters). Grab samples are then placed into an appropriate bucket (see the specifications for organic and/or inorganic parameters). Buckets are to remain sealed between grab sampling.

Once all grab samples have been taken and placed in the bucket, its contents are adequately homogenized with equipment matching the requirements of the chemical parameters to be analyzed. Disposable or veterinary gloves can be used for homogenization (see section 3 specifications for organic and/or inorganic parameters).

If a sample volume is less than or equal to 10 litres, a fraction of one litre of the contents is withdrawn and transferred to a prelabelled container that is appropriate for the chemical parameters to be analyzed (see the specifications for organic and/or inorganic parameters respectively described in 3.1 or 3.2). The container is now ready for shipment to the laboratory using the method described in section 3.4. Sample volume must be adjusted when plant matter is collected for the analysis of foreign matter.

[3] This is the minimum sampling volume when no other requirements need to be met. Two-litre sampling is required for foreign matter, and five-litre for plant matter. The capacity of all other containers must be adjusted on the basis of these volumes.

If the volume of the sample exceeds 10 litres, the quarterage technique is used to divide it (see section 3.1.4 or 3.2.4 for the description of the quarterage technique that is appropriate for the desired analysis). The composite sample thus obtained is transferred to a previously labelled sealable container that is appropriate for the planned chemical parameter analysis (see the specifications for organic and/or inorganic parameters respectively described in 3.1 or 3.2) and shipped to the laboratory using the method described in section 3.4.

3.4 Shipping samples

The method for shipping samples must be selected prior to commencing the sampling campaign. All samples must be properly packaged to ensure their integrity. A properly insulated cooler must be used and adjusted on the basis of the number, volume and position of the coolants with respect to the number, mass and initial temperature of the samples, in order to keep them cold. In all cases, care must be taken to ship the samples to the laboratory with all due haste.

4. Sampling for microbiology parameter analysis

Spot samples are collected from the production chain for continuously produced FRM. When FRM are discharged into more than one outlet channel, all channels must be sampled in equivalent manner. Samples for microbiology analysis should be taken during the final hour of the 8-hour sampling period in order to minimize conservation time between collection and analysis, which must never exceed 48 hours). Samples may be collected earlier in the day if the goal is to ship them to the laboratory before the other samples.

For batch FRM, grab samples are taken in the pile and used to prepare a composite sample.

For the salmonella parameter, and in order to check for the "zero presence" criterion in the sample, three different samples must be analyzed. This requirement must also be met if no salmonella is observed in at least two of the three analyzed samples.

4.1 Microbiology parameter analysis of continuously produced FRM

4.1.1 Required supplies

- Sterile polyethylene or similar resistant material, 65 or 75 microns in thickness, minimum one-litre sample bags with an integrated closure feature, preferably wide-mouthed and 20 cm in width by 30 cm in length, with sufficient available space for hermetical sealing;
- Disposable latex, nitrile or similar gloves;
- 70% ethanol or isopropanol (optimal concentration for disinfection) or individually packaged 70% ethanol or isopropanol swabs. An over-the-counter solution of 85% denatured ethanol and 15% methanol diluted with distilled water may also be used, as long as the final concentration of ethanol or isopropanol is 70%;
- An alcohol-resistant plastic sprayer bottle;
- Clean absorbent paper towels;
- A lidded plastic container reserved for disinfected matter;
- Frozen freezer packs;
- A cooler whose volume is sufficient to contain the sample and the appropriate number of freezer packs;
- Soapy water, rinse water (optional), distilled water and clean rags;
- Any other sampling supplies (clean spoon or ladle, etc.), as needed.

Sampling supplies must be stored at a clean location or kept in a contamination-free container.

4.1.2 Cleaning and disinfection

All reusable supplies (containers, spoons, etc.) must be washed with soapy water, rinsed with tap water (optional) then rinsed again with distilled water. Drying can be in the open air or by means of clean absorbent paper towels. Soapy water washing and distilled water rinsing can be handled prior to arrival on the sampling site if the clean supplies are transported in a clean, contamination-free container. Supplies may be washed and rinsed with distilled water on-site if there is an appropriate area for this in the plant itself.

Disinfection with alcohol must take place at the sampling site. Immediately before handling supplies, samplers must wash their hands with soapy water, and dry them. If soap and water are not available, hands

may be sprayed with a 70% ethanol solution or swabbed with an alcohol or disinfectant solution. Gloves must then be put on and disinfected by spraying with 70% ethanol. The inside and outside of the container reserved for disinfected supplies must also be disinfected, as well as all tools used for collecting samples, such as spoons. Sample bag exterior surfaces must also be disinfected by spraying with 70% ethanol or swabbed with an alcohol preparation. Bags should not be opened in order to remain sterile.

All other tools used for sampling should be dealt with in the same way.

Disinfected supplies are left to dry for at least one minute in the container reserved for this or at a previously disinfected location that is protected from airborne contamination. At the end of the one-minute waiting period and to accelerate drying, clean paper towels can be used. It is important that all supplies that come into contact with the substrate to be sampled are dry, otherwise analytical results may be distorted.

After disinfection and contact with any non-disinfected surfaces other than the matter to be sampled, all supplies must be disinfected again.

4.1.3 Labelling supplies

Bags are to be labelled after disinfection or immediately after sampling with a label whose number identifies the withdrawal point. If indelible ink is used, bags may be labelled prior to disinfection.

4.1.4 Sampling method

When samplers are ready to proceed, they put on gloves and disinfect them by spraying or swabbing with alcohol. In order to optimize disinfection, the gloved hands are rubbed together until they are completely dry. This is an important step, since it distributes the alcohol over the entire surface area of the gloves. Nothing should be subsequently touched except the sampling bags and supplies that were treated with disinfectant. A FRM sample of approximately one litre is then taken securely by hand or with a dry, disinfected tool, and placed in the bag, which is immediately sealed.

The FRM are then uniformly spread out inside the bag, taking care to reduce particle volume as much as possible so as to provide maximum surface contact for cooling. Once the bag has been filled and sealed, it must be clearly labelled if this was not previously done. Labelling must identify the sampling point.

The bag is immediately placed between two freezer packs. Immediately prior to or right after placing the bag in the cooler, the time is noted in the sampling log. When more than one sample is taken, the temperature in the cooler must be noted in the sampling log every hour or whenever a new sample is added to the cooler, until the time of shipment to the laboratory.

4.2 Batch FRM sampling for microbiology parameter analysis

4.2.1 Required supplies

- Sterile polyethylene or similar resistant material, 65 or 75 microns in thickness, at least one-litre sample bags with an integrated closure feature, preferably wide-mouthed and 20 cm in width by 30 cm in length, with sufficient available space for sealing hermetically;
- Disposable latex, nitrile or equivalent disposable gloves;
- 70% ethanol or isopropanol (optimal concentration for disinfection) or individually packaged 70% ethanol or isopropanol swabs. An over-the-counter solution of 85% denatured ethanol and 15% methanol diluted with distilled water may also be used, as long as the final concentration of ethanol or isopropanol is 70%;
- An alcohol-resistant plastic sprayer bottle;

- Clean absorbent paper;
- A lidded plastic container reserved for disinfected matter [4];
- A clean plastic lidded bucket, approximate capacity 20 litres;
- A clean spoon or ladle;
- Plastic sheeting;
- A plastic scoop;
- Frozen freezer packs;
- A cooler whose volume is sufficient to contain the sample and the appropriate number of freezer packs;
- A thermometer (for more than one composite sample);
- Soapy water, rinse water (optional), distilled water and clean rags.

Sampling supplies must be stored at a clean location or kept in a contamination-free container.

4.2.2 Cleaning and disinfection

All reusable supplies (buckets, spoons, scoops, veterinary gloves, etc.) must be washed with soapy water, rinsed with tap water (optional) then rinsed again with distilled water before departure for the sampling site. Clean absorbent paper towels can be used to dry the supplies, which are then transported in clean containers. Alcohol disinfection should take place on the sampling site, immediately prior to handling the supplies.

Once at the sampling site and before further disinfection of supplies takes place, samplers must wash their hands with soapy water, then dry them. If soap and water are not available, 70% ethanol may be sprayed, alcohol swabs applied or disinfectant solution used before putting on gloves and disinfecting them by spraying a 70% ethanol solution. The inside and outside of the container reserved for disinfected materials and the bucket (including the handle) are then disinfected, as well as all tools (pails, spoons, etc.) used for sampling. The outside of the sample bags should be disinfected by spraying a 70% ethanol solution or wiping with alcohol swabs. Bags must not be unsealed, to remain sterile.

All other tools used during sampling must be treated in the same way.

Disinfected supplies, except for the bucket, are left to dry for at least one minute in the container reserved for this or at a previously disinfected location that is protected from airborne contamination. At the end of the one-minute waiting period and to accelerate drying, clean paper towels can be used. It is important that all supplies that come into contact with the substrate to be sampled are dry, otherwise analytical results may be distorted.

After supplies have been disinfected they must, if they have been in contact with any surfaces other than the sampled matter, be disinfected again.

[4]A container that is reserved for disinfected supplies is not mandatory if the sampler possesses the means to protect them from microorganisms other than those found in the sampling pile.

4.2.3 Labelling supplies

Buckets must be labelled after disinfection and prior to sampling unless ethanol-resistant labelling ink is available. The numbering on the label must identify the sampling point and the bucket used must bear the same number as the bag sent for analysis.

Bags are labelled after disinfection or immediately after sampling and numbering must identify the sampling point. Bags may also be labelled prior to disinfection with indelible ink.

4.2.4 Sampling method

At least 10 grab samples are needed for the composite sample for piles whose volume is less than 400 m³. If volume exceeds that figure, the appropriate number of samples is determined by the following formula:

$$n = \frac{\sqrt{V}}{2}$$

where n is the number of samples and V is the volume in cubic metres (m³). The number of samples should not exceed 30.

The 30-sample maximum applies to piles with a maximum volume of 3,600 m³. If the pile has a volume greater than 3600 m³, it must be split evenly into sections, theoretically or virtually (not physically). The determination of the right number of grab samples uses the same formula for each section. For example, for a 5,000 m³ pile, the pile is divided into two separate 2,500 m³ (< 3,600 m³) sections. Twenty-five (25) grab samples must be taken to prepare a composite sample for each of the sections. In this example, two composite samples are to be analyzed.

When samplers are ready to proceed, they must put on gloves and disinfect them by spraying or swabbing with alcohol. In order to optimize disinfection, the gloved hands are rubbed together until they are completely dry. This is an important step, since it distributes the alcohol over the entire surface area of the gloves. Nothing should be subsequently touched except for disinfected supplies.

Sampling must be representative of the pile. As such, the entire pile must be covered by dividing it into a number of sections that correspond to the previously determined number of grab samples. Sampling volume must be sufficient to assess the characteristics of the matter to be recovered. Once the sampling columns are dug and satisfactorily represent the pile, they may be used to collect samples.

Grab samples must be identical and at least between .5 and 1 litres in volume, with depth varying between 30 centimetres and one metre, alternating between the top, middle and bottom of the pile. Grab samples may be taken using a disinfected plastic spoon or scoop. For samples taken with gloved hands, the sampler must be able to demonstrate that all column samples were identical in volume. Grab samples are placed into the disinfected bucket. Bucket lids must be used between sampling.

Once all grab samples have been collected and placed in the bucket, the contents can be appropriately homogenized with a tool or by hand, in the latter case wearing disinfected veterinary gloves to avoid sample contamination (see section 3). Approximately one litre of the contents is then withdrawn and transferred to an appropriate sterile bag, which is immediately sealed.

The FRM are then uniformly spread out inside the bag, taking care to reduce particle volume as much as possible so as to provide maximum surface contact for cooling. Once the bag has been filled and sealed, it must be clearly labelled at once. Labelling must identify the sampling point.

If the volume of the sample exceeds 10 litres, the quarterage technique is used to divide it. This involves emptying out the contents of the bucket onto a disinfected surface. A pile of regular shape is then made using a disinfected scoop, and divided into four. Two opposing quarters are then discarded and the remaining two combined. The process is repeated until a composite sample of the desired size is obtained. The composite sample is then transferred into a sterile sampling bag which is or will be properly labelled.

The FRM are then uniformly spread out inside the bag, taking care to reduce particle volume as much as possible to provide maximum surface contact for cooling. Once the bag has been filled and sealed, it must be clearly labelled if this was not previously done. Labelling must identify the sampling point. The time of day is noted in the sampling log and the bag immediately placed between two freezer packs in the cooler.

When more than one composite sample is taken, the temperature in the cooler must be noted in the sampling log every hour or whenever a new sample is added until the time of shipment to the laboratory.

4.3 Shipping samples for microbiology parameter analysis

The method for shipping samples must be selected prior to commencing the sampling campaign. It is strongly recommended to provide the laboratory with advance notification of incoming samples for microbiology analysis. All samples must be properly packaged to ensure their integrity and, to the extent possible, protect them from light. A properly insulated cooler must be used and adjusted on the basis of the number, volume and position of the coolants with respect to the number, mass and initial temperature of the samples, in order to cool them. In all cases, care must be taken to ship the samples to the laboratory with all due haste: for example, by messenger, and within an hour when possible.

5. Duplicates

A duplicate is a secondary or sub-sample from a spot or composite sample, distinct from the original, and taken for control and quality assurance purposes.

A duplicate created during sampling is used to demonstrate consistency of method and sampling equipment. The sample can be shipped as a ghost sample (identifying information not shared with the laboratory). It must be representative of the original and be labelled differently. Requesting the lab to perform two analyses from the same sample container does not constitute duplication.

A duplicate is a sub-sample from a single homogenized spot or composite sample. For composite samples, the bags or jars must be filled in rotation until the required volumes have been reached. In other words, a volume of the composite sample is transferred to the sample container, then an equivalent volume is transferred to the duplicate container, and so forth and so on in rotation until both containers have been filled. If the composite sample required quarterage, the final two opposing quarters must be used to prepare the duplicate: one quarter going into the sample container and the opposing quarter into the duplicate container.

When sampling continuously produced FRM, the duplicate used to test for *E. coli* must be an independent spot sample withdrawn identically and in the same period of time as the original. For salmonella, this step will be repeated three times in order to collect three distinct samples under identical conditions.

Duplicates must be prepared on at least 10% of total samples for inorganic chemistry, microbiology (*E. coli*) and foreign matter analysis. As such, the sampling log, must show a duplicate for every 10 samples and for each of the tests described herein.

Duplicates for chemical parameters must be tested for at least the following metals: copper, cadmium, chrome, zinc, and lead. As mentioned in DR-12-MRF, maximum acceptable variance among duplicates should not exceed 50%. Whenever this figure is exceeded, an explanation must be provided in the sampling report if the value is justifiable and accepted, and a non-compliance situation noted in the management system.

Microbiological duplicates can only be used to test for *E. coli*. Triplicate analysis is required for Salmonella to confirm whether or not it is present in at least two samples out of three.

Bibliography

AGENCE CANADIENNE D'INSPECTION DES ALIMENTS. Méthodes d'échantillonnage pour les engrais (T-4-114). Gouvernement du Canada, 1997, 8 pp. (English version untraceable).

BUREAU DE NORMALISATION DU QUÉBEC. *Organic Soil Conditioners–Composts* (CAN/BNQ 0413-200), 2016, 58 pp.

CENTRE D'EXPERTISE EN ANALYSE ENVIRONNEMENTALE DU QUÉBEC. *Programme d'accréditation des laboratoires d'analyse: normes et exigences* (DR-12-PALA). Québec, Ministère du Développement durable, de l'Environnement et des Parcs, 2012, 77 pp. (Available in French only).

CENTRE D'EXPERTISE EN ANALYSE ENVIRONNEMENTALE DU QUÉBEC. *Processus et exigences d'accréditation - Matières résiduelles fertilisantes - Secteur agricole (DR-12-MRF)*. Ministère de l'Environnement et de la Lutte contre les changements climatiques, 2019, 22 pp. (Available in French only).

CENTRE D'EXPERTISE EN ANALYSE ENVIRONNEMENTALE DU QUÉBEC. *Programme d'accréditation d'échantillonnage environnemental* (DR-12-PAÉE). Ministère du Développement durable, de l'Environnement et de la Lutte contre les changements climatiques, 2015, 15 pp. (Available in French only).

HÉBERT, Marc. *Guide sur le recyclage des matières résiduelles fertilisantes: Critères de référence et normes réglementaires*. Québec, ISBN-978-2-550-72954-9, 2015, 216 pp. (Available in French only).

Ontario Ministry of Agriculture, Food and Rural Affairs and Ministry of the Environment, Conservation and Parks. *Sampling and analysis protocol.* The techniques described are intended to meet the proper sampling and analytical requirements of Ontario Regulation 267/03 under the *Nutrient Management Act, 2002.* 2012, 16 pp.

MINISTÈRE DU DÉVELOPPEMENT DURABLE, DE L'ENVIRONNEMENT ET DES PARCS. *Sampling Guide for Environmental Analysis, Booklet 5, Soil sampling. May 2007(Revised, April 24, 2009)*. Centre d'expertise en analyse environnementale du Québec, 2010, 57 pp. and appendices.

MINISTÈRE DU DÉVELOPPEMENT DURABLE, DE L'ENVIRONNEMENT ET DES PARCS. *Sampling Guide for Environmental Analysis, Booklet 1–Overview*. Centre d'expertise en analyse environnementale du Québec, 2008, 58 pp.

Appendix I

Pathogen preventive measures for workers handling category P2 FRM

Preventive Measures	
Vaccination	• Usual immunization program applicable to the population as a whole
Protective equipment	• Latex or nitrile disposable gloves • Overalls or a disposable jumpsuit • Boots or overshoes • Face shield (when required by the type of work) • Waterless volatile antiseptic soap or disposable wipes • A first aid kit nearby that complies with the requirements of the First-aid Minimum Standards Regulation (CQLR c A-3.001, r 10)
Sanitary measures	• Wear clean work clothing • Avoid wiping eyes and mouth or touching the face • Wash hands frequently during the day per CLSC instructions, especially before eating, drinking or smoking • Trim fingernails • Never keep food, beverages or tobacco in work suit pockets • Avoid sampling during high wind conditions that can cause bioaerosol drift • Disinfect cuts or skin lesions and apply protective covering to prevent all contact with residues • Wash all clothing and equipment that has been in contact with category P2 FRM (boots and vehicle wheels, running boards and floors, etc.) • Remove all soiled work clothes before leaving for home, placing them in a plastic bag and notifying the person in charge of washing them • Shower and shampoo at work at the end of the day

Appendix II

Sampling Data Log Template

Month _____ **Year** _____ **Sampling location** _____

Sampling method[1] _____ **Pile volume**_____

Sample number	Protective equipment	Date	Cooler (Temp. °C)	Time	Types of residue					Types of analysis[3]				Remarks[4]	Sampled by
					D	P	P/S	M	C	OC	IC	M	FM		

[1]Sampling method used (adapted per the *Protocole d'échantillonnage de matières résiduelles fertilisantes et dispositions particulières liées à l'accréditation*, DR-12-MRF-02).

[2]Type of residue: D = de-inked; P = primary; P/S = primary-secondary; M = mixed (P/S + D or other–describe the mix); C = cinders.

[3]Type of analysis: OC = organic chemistry; IC = inorganic chemistry; M = microbiology; FM= foreign matter.

[4]Describe all anomalies or particular details that could affect sample quality and log all changes to the sampling method.

Environnement
et Lutte contre
les changements
climatiques
Québec